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Field Testing of High Current Electrokinetic Nanoparticle Treatment for Corrosion Mitigation in Reinforced Concrete

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This work examines field performance of nanoscale pozzolan treatments delivered electrokinetically to suppress chloride induced corrosion of concrete reinforcement. The particles are 20 nm silica spheres coated with 2 nm alumina particles that carry a net positive charge. Earlier work demonstrated that the alumina particles were stripped from the silica carriers and formed a dense phase with an interparticle spacing that is small enough to inhibit the transport of solvated chlorides. A D.C. field was used to inject the particles into the pores of concrete specimens, directly toward the mild steel bars that were embedded within each 3 inch diameter by 6 inch length concrete specimen. The voltage was held constant at 25 v per inch of concrete cover for a period of 7 days. These voltages permitted current densities as high as 3 A/m^2 . During the final 3 days, a 1 molar solution of calcium nitrate tetrahydrate was used to provide a source of calcium to facilitate stronger and more densified phase formation within the pores. In a departure from prior work the particle treatments were started concurrent with chloride extraction in order to determine if particle delivery would inhibit chloride transport. Following treatment the specimens were immersed in seawater for 4 weeks. After this post-treatment exposure, the specimens were tested for tensile strength and the steel reinforcement was examined for evidence of corrosion. Scanning electron microscopy was conducted to assess impact on microstructure.



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Overview

- Background
 - Nanoparticle
 - Rebar Corrosion
- Experimental Design
 - Specimen
 - Treatments
 - Electrokinetic Nanoparticle (EN)
 - EN with additional Calcium treatment (EN + Ca)
 - Electrochemical Chloride Extraction (ECE)
 - Post-treatment exposures



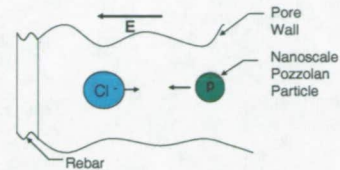
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Nanoparticle Treatment

- Nanoparticle used was alumina coated silica which carries a positive charge
- Nanoparticle size: 24 nm (20 nm silica interior surrounded by 2 nm layer of alumina)
- Nanoparticles predicted to form barrier surrounding rebar which will prevent chlorides from attacking rebar

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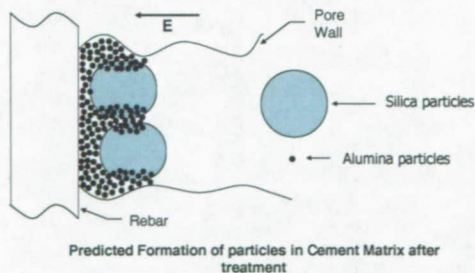
Concept of Nanoparticle Treatment



Reactive Electrokinetic Treatment in Cement Matrix

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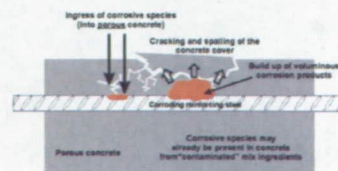
Concept of Nanoparticle Treatment



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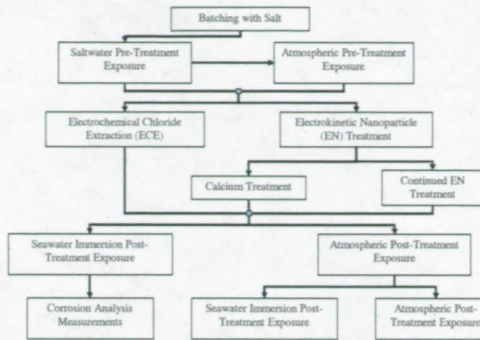
Rebar Corrosion

- Causes of Corrosion
 - Cl⁻ catalyzed attack by dissolved oxygen
 - Drop in concrete pH depassivates rebar



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Experimental Flow Chart



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Specimen Design

- 3" Dia. by 6" Ht.
- Reinforced with 0.25" Dia. 1018 steel
- Sealed exposed rod and top of concrete with red epoxy

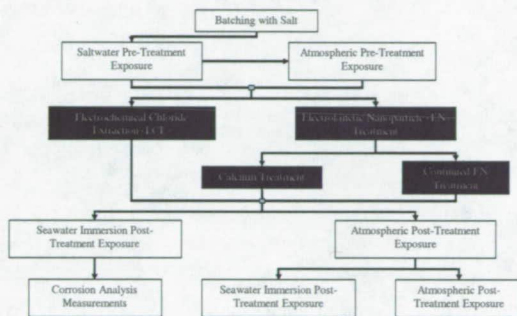


Batch Composition

Materials	Weight (lbs)
Water	18.5
Cement	36.5
Gravel	93.5
Sand	56.5
Salt	0.83

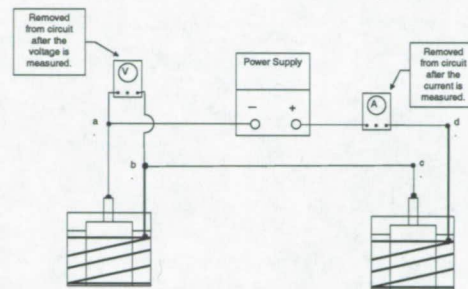
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Treatments



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Treatment Setup



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Treatment Procedure

- Two specimens per power supply (one EN and one ECE)
- Treatment voltage: 37.5 V (25 V per in. of concrete cover)
- Current Density < 10 A/m²
- Voltage & Current checked daily



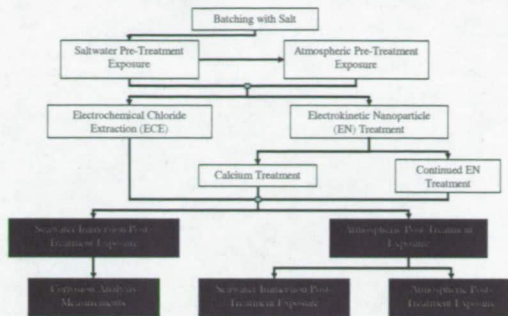
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Treatment Types

- Electrokinetic nanoparticle (EN)
 - Treatment duration: 7 days
- Electrokinetic nanoparticle plus additional introduction of calcium (EN + Ca)
 - Treatment duration: 4 days of EN and 3 days of Calcium
- Electrochemical Chloride Extraction (ECE)
 - Treatment duration: 7 days

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Post-treatment Exposures



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Post-Treatment Exposure



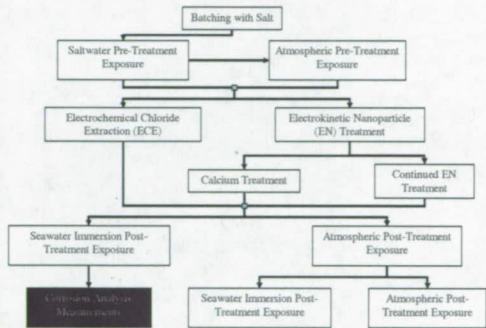
Seawater Immersion



Atmospheric Exposure

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Results



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Corrosion Measurements

- Corrosion potential (V_{corr})
 - Measured daily during treatment and weekly thereafter
- Corrosion Rate
 - Linear Polarization Resistance (LPR) method
 - Measured after 1, 4, and 7 days of treatment
- Corroded Area Coverage
 - Breaking apart specimen and visually

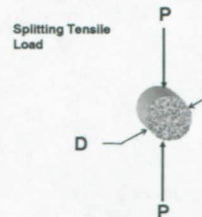
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Other Analyses

- Indirect Tensile Test
- Scanning Electron Microscopy (SEM)
 - Fractured sample
 - Polished sample (for elemental composition via EDAX¹)

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Indirect Tensile Test

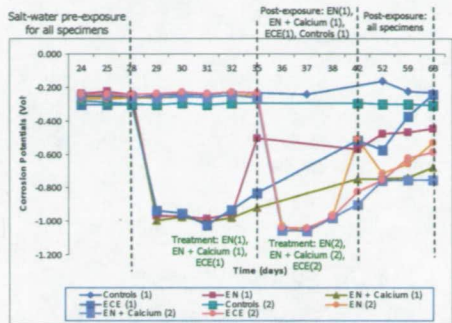


σ = Tensile Strength
 P = Load Applied
 L = Length
 D = Diameter

In Accordance with ASTM C 496-96

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Corrosion Potential



All corrosion potentials measured with respect to S.H.E.

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Linear Polarization (immersion only)

Treatment Type	Corrosion rate (mpy)		
	After 1 Day of treatment	After 4 days of treatment	28 days post-treatment
Controls	0.4 ¹		2.7 ²
EN	1.7	0.5	3.5
EN + Ca	1.5	0.6	1.6
ECE	2.5	2.2	3.8

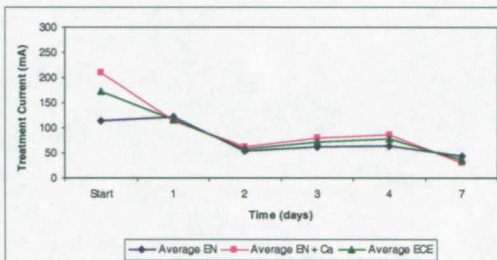
¹ Taken day treatment was started

² 0.5 mpy is considered serious corrosion in steel concrete reinforcement

- Corrosion rate for control specimens can be interpreted as the corrosion of the rebar
- Corrosion rate for treated specimens does not represent corrosion of steel

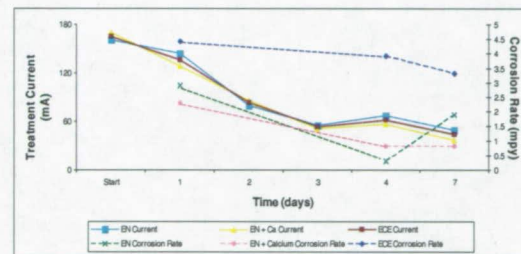
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Treatment Current for Atmospheric Specimens



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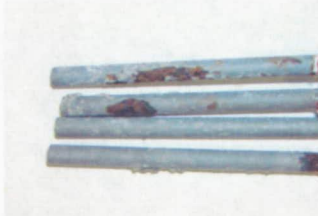
Average Treatment Current vs. Corrosion Rate



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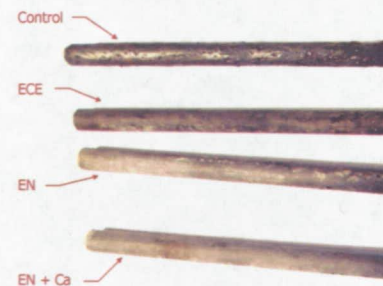
Pre-treatment Corrosion

- Average percent area corroded: 3% $\pm$ 1%
- Corrosion rate: 0.41 mpy $\pm$ 0.43 mpy
- Considered serious corrosion for steel reinforcement when 0.46 mpy



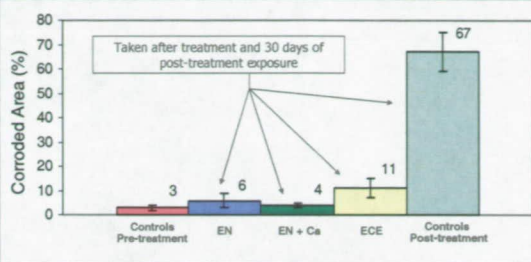
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Corroded Area (post-treatment)



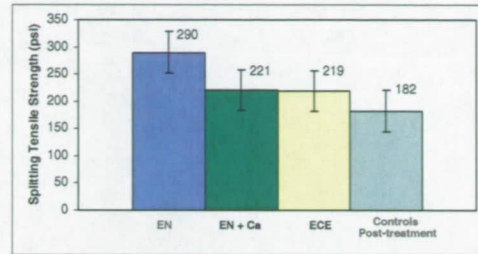
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Corrosion Product Analysis



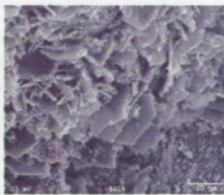
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Splitting Tensile Strength Test Results

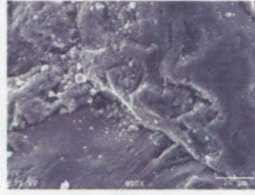


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Microstructure Analysis



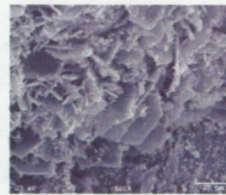
Control



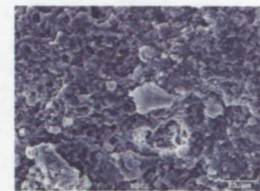
EN Specimen

Images taken 1 mm from rebar

Microstructural Analysis



Control

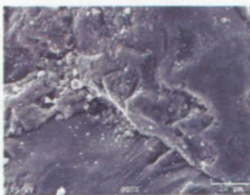


ECE Specimen

Images taken 1 mm from rebar

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Comparison of EN and EN + Ca



EN Specimen



EN + Ca Specimen

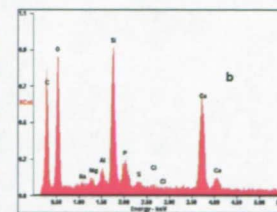
Images taken 1 mm from rebar

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EDAX for EN Treated Specimen



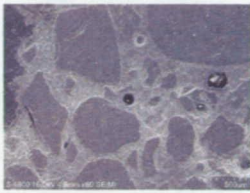
a) Polished Section



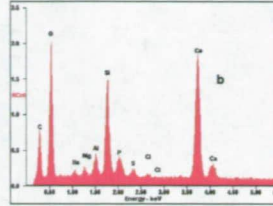
b) EDS Analysis

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EDAX for EN + Ca Treated specimen



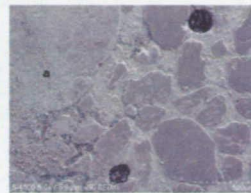
a) Polished Section



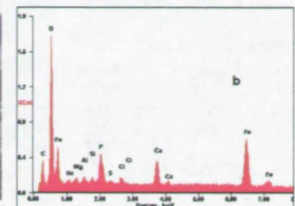
b) EDS Analysis

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EDAX for ECE Treated Specimen



a) Polished section



b) EDS Analysis

Elemental Compositions via EDAX

Specimen Type	Elements (Wt%)				
	Chlorine	Sodium	Aluminum	Calcium	Silica
Controls	4.1	3.2	1.0	10.9	23.8
EN	0.0	0.3	1.1	14.8	10.0
EN +Ca	0.4	0.5	1.7	23.7	5.6
ECE	0.8	0.5	0.6	8.5	1.0

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Conclusions

- High current used during treatment supplied enough force to deliver particles in less than a week
- All treatments were able to mitigate corrosion in a short period of time, a more extensive analysis in the future could produce better results
- Introduction of calcium into specimen did not have as much effect on strength as predicted; although, it did not have a negative effect on corrosion mitigation

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Acknowledgements

- Paul Hintze
- Mark Kolody
- Jerry Curran
- Wendy Li
- Teddy Back
- Jerry Buhrow

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